

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
14 June 2001 (14.06.2001)

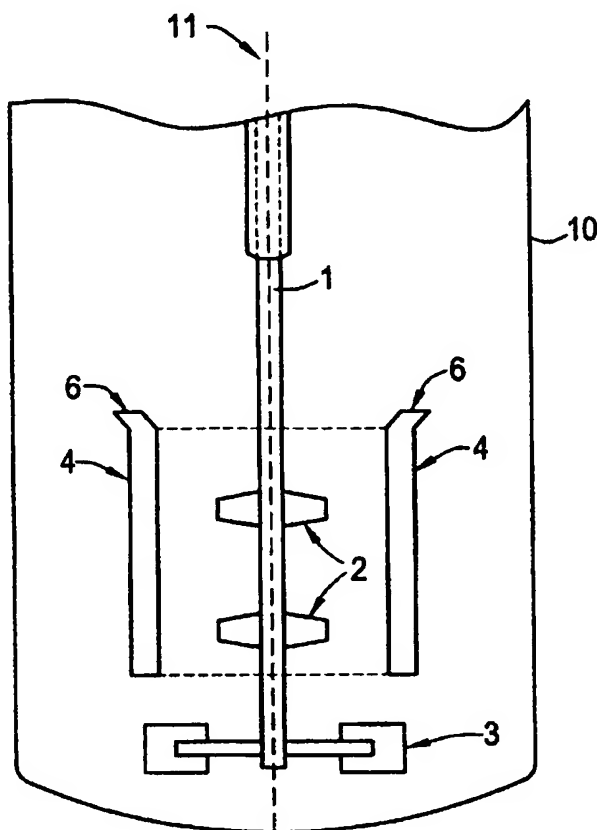
PCT

(10) International Publication Number  
**WO 01/41919 A1**

- (51) International Patent Classification<sup>7</sup>: **B01J 19/18**, 19/20, B01F 7/16, 7/22
- (21) International Application Number: **PCT/US00/32586**
- (22) International Filing Date:  
30 November 2000 (30.11.2000)
- (25) Filing Language: **English**
- (26) Publication Language: **English**
- (30) Priority Data:  
60/170,053 10 December 1999 (10.12.1999) **US**
- (71) Applicant (for all designated States except US): **INCA INTERNATIONAL S.P.A.** [IT/TT]; Via Patroclo, 21, I-20151 Milan (IT).
- (72) Inventors; and
- (75) Inventors/Applicants (for US only): **KAR, Kishore** [US/US]; 4616 Oakridge Drive, Midland, MI 48640 (US). **PIRAS, Luciano** [IT/TT]; Via Roberto Bellarmino, 1, I-20141 Milan (IT).
- (74) Agent: **JOZWIAK, Elisabeth, T.**; Intellectual Property, P.O. Box 1967, Midland, MI 48641-1967 (US).
- (81) Designated States (national): **AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.**
- (84) Designated States (regional): **ARIPO** patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), **Eurasian**

[Continued on next page]

(54) Title: **IMPELLER DRAFT TUBE AGITATION SYSTEM FOR GAS-LIQUID MIXING IN A STIRRED TANK REACTOR**



(57) Abstract: The present invention relates to design for the agitation system in stirred tank reactors for gas-liquid reactions. In particular the present invention relates to a system which incorporates a draft tube, one or more axial impellers and one or more radial impellers. The agitation design offers contact between a liquid phase and a gaseous phase within the reactor.



WO 01/41919 A1



patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

**Published:**

— *With international search report.*

## IMPELLER DRAFT TUBE AGITATION SYSTEM FOR GAS-LIQUID MIXING IN A STIRRED TANK REACTOR

The present invention relates to an improved design for the agitation system in stirred tank reactors for gas-liquid reactions. In particular the present invention relates to a system which incorporates a draft tube, one or more axial impellers and one or more radial impellers. The new agitation design offers improved contact between a liquid phase and a gaseous phase within the reactor.

Stirred Tank Reactors (STRs), in which gas and liquid phases make intimate contact for mass transfer, are very common in chemical processes such as fermentation, hydrogenation, phosgenation, neutralization, chlorination, and organic oxidation. The design of the STR has a significant effect on gas bubble dispersion, interfacial area ("a"), bubble surface transience, and the mass transfer coefficient " $K_L$ ". These factors in turn effect the rate of conversion, selectivity and the yield of the reaction. The design of the STR also has an impact on the power needed to run the impeller system at a given rate.

Many different mixing systems have been reported in the art to try and achieve the maximum mass transfer between the gas and liquid phases. These include U.S. Patents 4,231,974; 5,108,662; 5,371,283; 5,451,349; 5,523,474; 5,536,875; 5,696,285. These prior mixing attempts can be improved, as they do not have optimal mass transfer coefficients nor have they minimized the power requirements.

The present invention incorporates a draft tube along with both axial impeller and radial impellers. The axial impeller(s) is (are) located inside the cylinder formed by the draft tube, while the radial impeller(s) is located below the cylinder formed by the draft tube. The fluid flow and the mass transfer characteristics of such a system are superior to the conventional agitation system.

Additional advantages and features of the present invention will become apparent from a reading of the detailed description of the invention which makes reference to the following drawing.

Figure 1 is a cross-sectional view of an apparatus which corresponds to the present invention.

Figure 2 is a graph of observed  $K_L a$  vs. gas flow rate for three different agitation rates in a system having a draft tube.

Figure 3 is a graph of observed  $K_L a$  vs. gas flow rate for three different agitation rates in a system not having a draft tube.

5 As seen in Figure 1, the present invention comprises a shaft 1; at least one axial impeller 2 attached to the shaft 1, for moving fluid in a direction generally parallel to the shaft's axis 11; at least one radial impeller 3 attached to the shaft 1, for moving fluid in a direction generally perpendicular to the shaft's axis 11; and a draft tube 4 in the shape of a generally open cylinder. As is clearly shown in Figure 1, when the agitation system 5 is  
10 placed within a stirred tank reactor 10, the shaft 1 extends through the draft tube 4 and the one or more axial impellers 2 are located within the draft tube cylinder 4 and the one or more radial impellers 3 are located outside of the draft tube cylinder 4.

The stirred tank reactor 10 with the agitation system 5, should be arranged in a vertical fashion as presented in Figure 1, such that the radial impellers 3 are located below  
15 the draft tube 4. Also as shown in Figure 1, the entrance to the draft tube 4 is preferably beveled away from the shaft 1, although this is not mandatory.

As the shaft 1 rotates around its axis 11, the axial impellers 2 act to move the reactor contents down through the draft tube 4 in a direction generally parallel to the shaft's axis 11. Axial impellers are generally known in the art and any such impellers may be used in the  
20 present invention. For example, a double helix impeller such as the one depicted in U.S. 5,108,662, or an airfoil blade impeller such as the one depicted in U.S. Patent 4,231,974 could be used in this invention. Other suitable axial impellers include Pitch Blade Turbine, high efficiency impellers (such as model A-310 from Lightnin Mixing Co, HE-3 from Chemineer, Inc. and Viscoprop from EKATO Rueher and Mischtechnik GmbH), single  
25 helixes or marine props (such as A-315 or A-320 from Lightnin Mixing Co., MT-4, or MY-4 from Chemineer, Inc.). The number of axial impellers used in general depends on the viscosity of the working media. The more viscous the working media the more axial impellers are warranted. It is contemplated that the invention may comprise from 1 to several axial impellers 2, but it is preferred that there be two.

Simultaneously, as the shaft 1 rotates around its axis 11, the radial impellers 3 act to move the reactor contents away from the shaft 1, and (as the radial impellers 3 are located below the draft tube 4) outside the draft tube 4. The center line of the radial impeller(s) should be far enough below the end of the draft tube to avoid substantial interference. This is typically in a range of from about 1/8 the distance of the radial impeller's diameter to about 7/8 of the radial impeller's diameter, with about 2/3 being most preferred. Radial impellers are also generally known in the art, and any design may be used in the current invention. Common radial impellers which are suited for use in the present invention include flat blade impellers, Rushton Impellers, Concave Disk Turbine (Smith turbine) SCABA (SRGT) impellers and model BT-6 from Chemineer, Inc. The optimum number of radial impellers to be used is dependent upon the ratio of the liquid height to the tank diameter. In most cases a single radial impeller will be used in the current invention, but in some reactors, such as tall fermenters, multiple radial impellers may be used.

Draft tubes and their modifications are also well known in the art, and those teachings are generally applicable to this invention. For example, the draft tube can be slotted to provide for return of liquid to the center of the draft tube if the level of liquid for some reason does not exceed the top of the draft tube. Also, the use of vertical baffles on the inner surface of the draft tube can be advantageously used to redirect tangential flow to axial flow. If baffles are used in the draft tube it is preferred that they have a width of 0.8 to 0.1 of the draft tube inner diameter with a clearance of 0.016 to 0.021 of the draft tube inner diameter. Moreover, the use of a baffle to partially close of the bottom of the cylinder formed by the draft tube is shown, inter alia, in U.S. Patent 5,536,875 and may also be used in the present invention.

Although the dimensions of the draft tube 4 are not critical to the present invention, it has been found that the optimum radius of the draft tube is 0.707 of the tank radius. Using a draft tube of this radius make the cross sectional area of the tank which is inside the draft tube equal the cross sectional area of the tank which is outside the draft tube. As seen in Figure 1, the draft tube 4 can optionally contain a conically flared portion 6, at the entrance end of the draft tube. It is believed that this section will aid in straightening the flow of the reactor contents. The angle of the bevel should be between 30 and 60 degrees, with 45 degrees being most preferred. The beveled edge should not be too long, such that it restricts

flow around the top of the draft tube. It is preferred if the length of the beveled edge is from zero to about one fourth of the draft tube's inner diameter, with about 1/12 of the length being most preferred.

5 The present invention can be used with stirred tank reactors of any dimensions. The draft tube can be held in the appropriate position using side structural braces which attach to the reactor wall, as is known in the art. Also along the reactor wall, baffles can be optionally used, as is generally known in the art. If used, there are preferably four baffles spaced approximately  $90^\circ$  apart from each other. The reactant gas can be brought into the tank by any apparatus known in the art. These include ring spargers and more preferably pipe or  
10 nozzle spargers.

It should be understood that specific details of construction of the invention, such as materials, dimensions and the like, are not to be considered as limitations of the invention. Rather, these details can be adjusted as needed to create a preferred embodiment of the invention for any particular application.

15 The effectiveness of the present invention may be seen in the following Examples:

#### EXAMPLES

A transient technique was used to determine the mass transfer coefficient. This dynamic gassing out technique consists of sparging the reactor with pure nitrogen until all oxygen has been stripped from the working media. The sparge gas is then rapidly changed  
20 from nitrogen to oxygen. The transient oxygen concentration is then measured. These measurements can then be used to calculate the volumetric mass transfer coefficient ( $K_L a$ ) for the system. Physical techniques such as this are applicable to a well mixed system and to small values of  $K_L a$  due to the slow response time of dissolved oxygen probes. In general these physical techniques are applicable where  $K_L a < 1/\tau$ , where  $\tau$  is the dissolved oxygen  
25 probe response time.

An ASME dish bottom PLEXIGLAS™ tank having a 0.45 m inner diameter ( $0.08\text{m}^3$ ) was used to conduct the gas-liquid mixing experiments. Four flat baffles spaced  $90^\circ$  apart, were used to facilitate axial mixing. The agitation system included two high-

efficiency down-pumping axial impellers (model A-310 from Lightnin Mixers Ltd.), and one radial gas dispersing Rushton disk turbine (Lightnin R-100). The gas was sparged through a ring sparger located below the radial impeller. Deionized water was used as the working media. The agitator speed and torque were measured by a proximity tachometer and load cell, respectively, while the concentration of dissolved oxygen in the water was measured with two oxygen sensors (Electrosense DO probes having a response time of 2 seconds for 95% saturation). The agitation levels (gassed power of 0.26 -2.6 watt/kg) were sufficient to create uniform gas dispersion at a 0.012 - 0.046 m/s (i.e. 1-5 VVM) superficial gas velocity. All of the data was collected using a CAMILE™ 2000 data acquisition system. The  $K_L a$  at different gas flow rates and shaft speeds (power) was first determined for the system without a draft tube. ANSI/ASCE Standard 2-91 entitled "Measurement of Oxygen Transfer in Clean Water" was used with non-linear regression (such as in the statistical software package known as JMP from SAS Institute, Cary, North Carolina, U.S.A.) to determine the correlation between the Mass transfer coefficient,  $K_L a$  (1/sec) and superficial gas velocity,  $V_{sg}$  (m/sec). These results are shown in Table 1 and graphically depicted in Figure 2.

**Table 1****Mass Transfer Coefficient without the Draft Tube**

	Speed [rpm]	Gas flow rate Q [scfm]	Power per mass $\epsilon$ [w/kg]	Superficial gas velocity $V_{sg}$ [m/s]	Mass transfer coefficient N $k_L a$ [1/sec]
20	300	4	2.66	0.0122	0.063
	300	6	2.30	0.0182	0.070
	300	9	2.14	0.0274	0.077
25	300	12	2.21	0.0365	0.081
	250	4	1.66	0.0122	0.057
	250	6	1.55	0.0182	0.058
	250	9	1.49	0.0274	0.068
	250	12	1.52	0.0365	0.072
30	250	15	1.49	0.0456	0.074
	200	4	1.06	0.0122	0.043
	200	6	1.02	0.0182	0.046
	200	9	0.95	0.0274	0.053
	200	12	0.96	0.0365	0.064
35	200	15	0.94	0.0456	0.067

Then a draft tube having an inner diameter of 0.3 m (to achieve approximately equal

superficial gas velocities in the draft tube and the annular region surrounding the draft tube) was placed in the mixing system. The  $K_L a$  was again determined at different gas flow rates and shaft speeds (power). The results with the draft tube are shown in Table 2 and depicted graphically in Figure 3.

5

**Table 2****Mass Transfer Coefficient with the Draft Tube**

	Speed N [rpm]	Gas Flow Rate Q [scfm]	Power per mass $\epsilon$ [w/kg]	Superficial gas velocity Vsg [m/s]	Mass transfer coefficient kLa [1/sec]
10	300	4	2.21	0.0121	0.075
	300	6	1.85	0.0182	0.081
	300	9	1.53	0.0273	0.094
	300	12	1.50	0.0365	0.099
	300	15	1.45	0.0456	0.099
15	250	4	1.28	0.0121	0.062
	250	6	1.08	0.0182	0.069
	250	9	0.90	0.0273	0.075
	250	12	0.85	0.0365	0.083
	250	15	0.88	0.0456	0.092
20	200	4	0.70	0.0121	0.045
	200	6	0.65	0.0182	0.051
	200	9	0.61	0.0274	0.059
	200	12	0.60	0.0365	0.063
	200	15	0.52	0.0456	0.069
25					

As seen from these results, the presence of the draft tube increases the  $K_L a$  over that of a conventional system having no draft tube, increases the gas hold-up and decreases the power draw for the same motor speed.

30

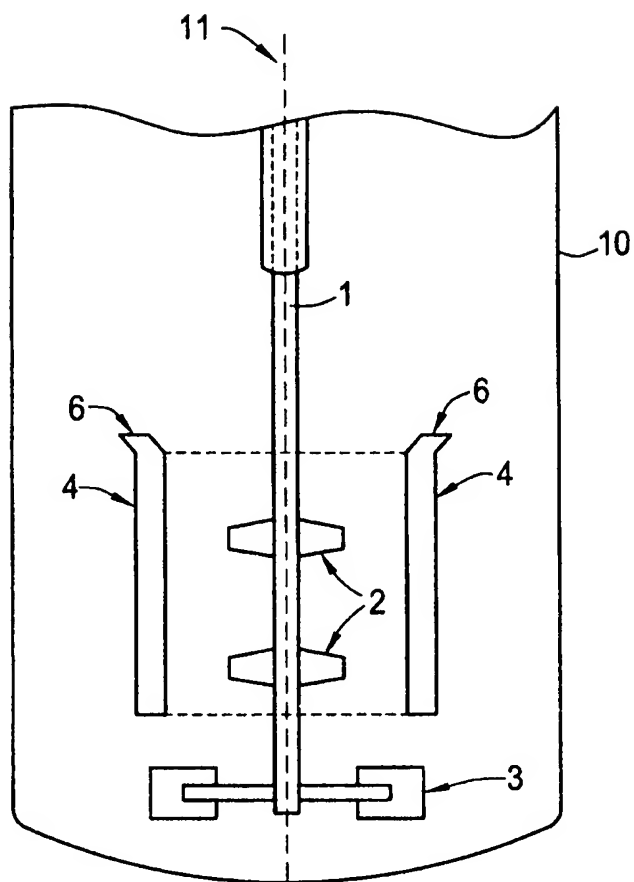


## WHAT IS CLAIMED IS:

1. An agitation system suitable for use in a stirred tank reactor, comprising:
  - a. a shaft
  - b. at least one axial impellar attached to the shaft, for moving fluid in a  
5 directions generally parallel to the shaft;
  - c. at least one radial impeller attache to the shaft, for moving fluid in a direction generally perpindicular to the shaft;
  - d. a draft tube in the shape of a cylinder, and having an opening at both ends of the cylinder;
- 10 wherein the shaft extends through the draft tube and wherein the axial impeller is located within the draft tube cylinder and the radial impeller is located outside of the draft tube cylinder.
2. The system of Claim 1 wherein the shaft is arranged vertically, and the radial impeller is located below the draft tube.
- 15 3. The system of Claim 2 wherein the draft tube further comprises a conically flared section at the top of the cylinder.
4. The system of Claim 2 wherein the axial impeller is in the form of a double helix.
5. The system of Claim 2 wherein the axial impeller is a high efficiency  
20 impeller.
6. The system of Claim 2 wherein there are two axial impellers.
7. The system of Claim 2 where in the draft tube contains slots to permit liquid to enter the inside of the draft tube without going over the top of the drat tube.

1/3

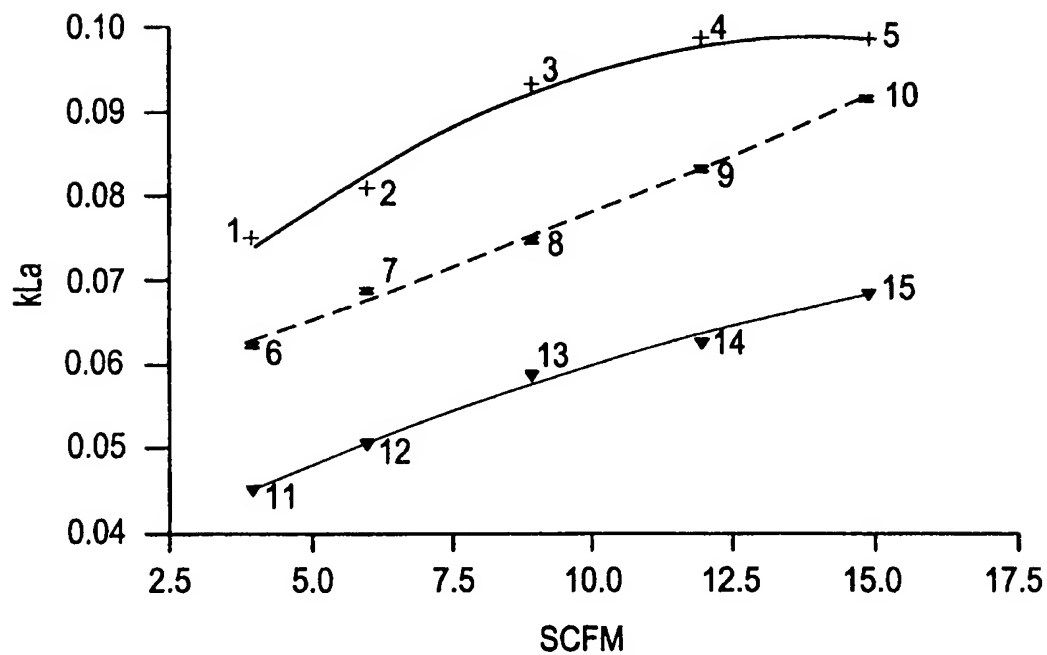
FIG. 1



2/3

## FIG. 2

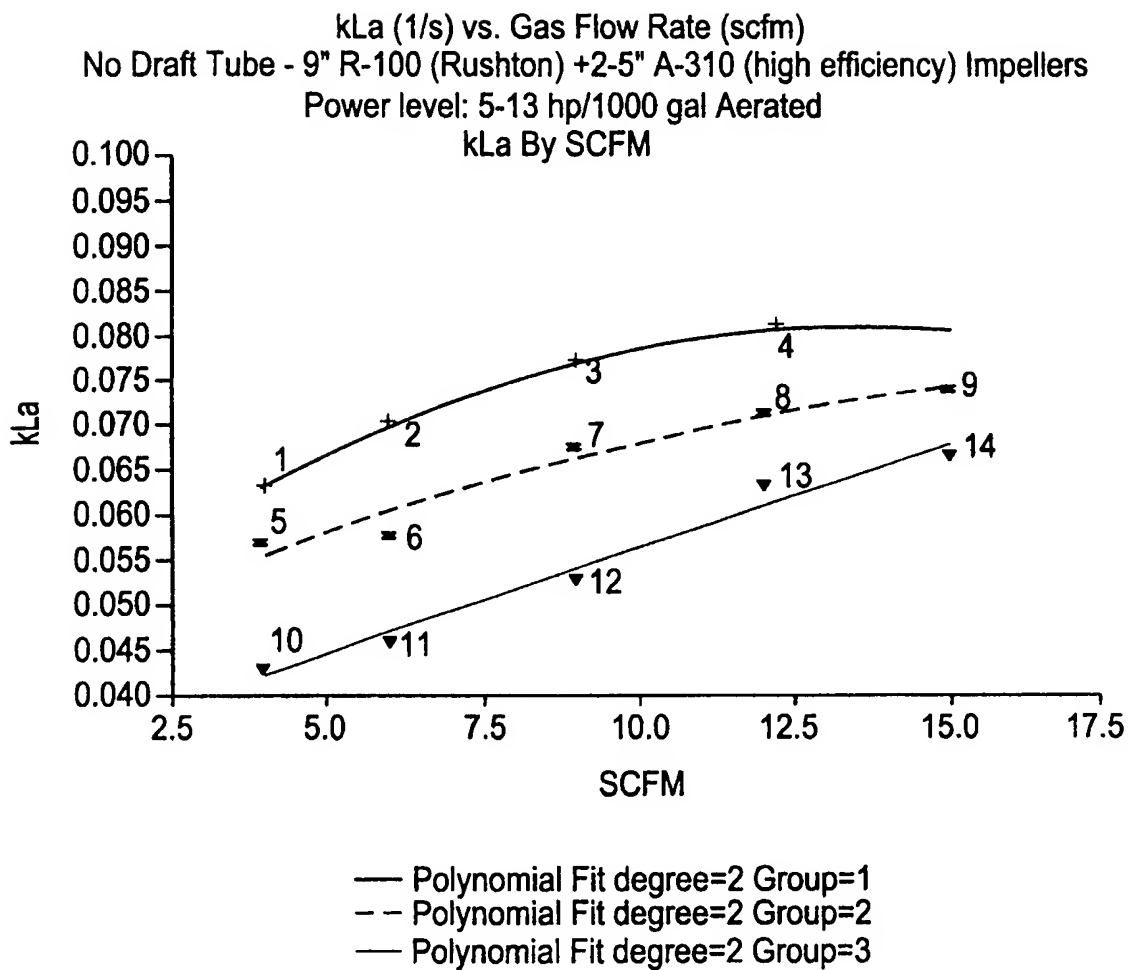
kLa (1/s) vs. Gas Flow Rate (scfm)  
With Draft Tube - 9" R-100+2-A-310 (high efficiency) Impellers  
Power level: 2.5-11 hp/1000 gal  
kLa By SCFM



— Polynomial Fit degree=2 Group=1  
-- Polynomial Fit degree=2 Group=2  
— Polynomial Fit degree=2 Group=3

3/3

FIG.3



# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 00/32586

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 B01J19/18 B01J19/20 B01F7/16 B01F7/22

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 B01J B01F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, EPO-Internal

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 972 661 A (PAUL M. KUBERA ET AL.) 26 October 1999 (1999-10-26) column 1, line 1 - line 9 column 2, line 26 - line 57 column 7, line 56 -column 8, line 21 figure 6 ----	1,2,5,6
X	US 4 699 740 A (FRANZ M. BOLLENRATH) 13 October 1987 (1987-10-13) column 2, line 29 - line 45 column 2, line 64 -column 3, line 40 figure 1 ----	1,2,5,6
X	US 3 460 810 A (HANS MUELLER) 12 August 1969 (1969-08-12) column 1, line 35 - line 41 column 3, line 24 -column 4, line 3 figure 1 ----- -/--	1,2,5-7



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

### \* Special categories of cited documents :

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

\*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

\*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

\*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

\*G\* document member of the same patent family

Date of the actual completion of the international search

20 March 2001

Date of mailing of the international search report

30/03/2001

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax: (+31-70) 340-3016

Authorized officer

Stevnsborg, N

# INTERNATIONAL SEARCH REPORT

In International Application No  
PCT/US 00/32586

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 97 25133 A (KVAERNER PULPING AB) 17 July 1997 (1997-07-17) page 3, line 22 -page 4, line 2 page 4, line 31 -page 5, line 14 figure 1 ---	1-3,5,6
X	US 4 798 131 A (NAOTAKE OHTA ET AL.) 17 January 1989 (1989-01-17) column 3, line 50 -column 4, line 25 figure 1 ---	1,2,5,6
X	US 4 388 447 A (MUNE IWAMOTO ET AL.) 14 June 1983 (1983-06-14) column 3, line 47 - line 52 figure 1 ---	1,2,5
Y	---	4
X	DATABASE WPI Section Ch, Week 198516 Derwent Publications Ltd., London, GB; Class J04, AN 1985-097623 XP002163361 & SU 1 115 791 A (VOLKOV I A), 30 September 1984 (1984-09-30) abstract; figure 1 ---	1,2,5
Y	---	4
X	DATABASE WPI Section Ch, Week 199145 Derwent Publications Ltd., London, GB; Class A41, AN 1991-331560 XP002163362 & SU 1 632 493 A (SHISHKIN A V), 7 March 1991 (1991-03-07) abstract; figure ---	1,2,5,6
Y	US 5 536 875 A (ANNE K. ROBY & JEFFREY P. KINGSLEY) 16 July 1996 (1996-07-16) cited in the application figure 2 -----	4

# INTERNATIONAL SEARCH REPORT

Information on patent family members

In International Application No

PCT/US 00/32586

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5972661 A	26-10-1999	AU 6267899 A WO 0018948 A	17-04-2000 06-04-2000
US 4699740 A	13-10-1987	DE 3516027 A EP 0200886 A JP 61257224 A	06-11-1986 12-11-1986 14-11-1986
US 3460810 A	12-08-1969	CH 486559 A CH 486560 A CH 489609 A BE 700543 A DE 1557185 A DK 131923 B FR 1588763 A GB 1162584 A JP 54024138 B NL 6709258 A,B SE 311343 B	28-02-1970 28-02-1970 30-04-1970 01-12-1967 25-02-1971 29-09-1975 16-03-1970 27-08-1969 18-08-1979 05-01-1968 09-06-1969
WO 9725133 A	17-07-1997	SE 505871 C BR 9706926 A SE 9600100 A US 6030113 A	20-10-1997 20-07-1999 13-07-1997 29-02-2000
US 4798131 A	17-01-1989	JP 1799983 C JP 5008673 B JP 62069976 A AT 64615 T AU 601160 B AU 6305286 A DE 3679878 D EP 0216702 A US 4891236 A	12-11-1993 02-02-1993 31-03-1987 15-07-1991 06-09-1990 26-03-1987 25-07-1991 01-04-1987 02-01-1990
US 4388447 A	14-06-1983	JP 1400609 C JP 57096006 A JP 62009245 B BR 8107906 A GB 2088884 A,B IN 156660 A IT 1140314 B KR 8900235 B MX 158872 A	28-09-1987 15-06-1982 27-02-1987 14-09-1982 16-06-1982 05-10-1985 24-09-1986 11-03-1989 27-03-1989
SU 1115791 A	30-09-1984	NONE	
SU 1632493 A	07-03-1991	NONE	
US 5536875 A	16-07-1996	BR 9501991 A CA 2149058 A CN 1112910 A EP 0682000 A JP 8059546 A KR 238501 B RU 2140898 C	27-02-1996 12-11-1995 06-12-1995 15-11-1995 05-03-1996 15-01-2000 10-11-1999